



Case HF/5-22102/A/PCT

Declaration

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE PCT NATIONAL STAGE APPLICATION OF      Group Art Unit: 1751

PETR KVITA ET AL

SERIAL NO.: 10/089,852

FILED: July 8, 2002

FOR: Improved Fabric Softener Compositions

DECLARATION UNDER RULE 132

I, Mario Dubini, a citizen of Switzerland residing in Niederdorf (Baselland),  
Switzerland hereby declare:

1. That I was awarded the Technical Laboratory Assistant in 1974 in Basel, Switzerland.
2. That I have been employed by Ciba Specialty Chemicals as a Technical Laboratory Assistant since April 1974.
3. That I presently hold the position of a Head of Application Laboratory Fabric Surface Modification in Grenzach, Germany.
5. That I consider myself an expert in the field of Textile dying, finishing, care, especially laundry processes.
6. That I prepared the test protocoll and performed the application tests of the compounds herein described in strict accordance with my statements in the Declaration.

### Hydrophilicity

The following test serves to demonstrate that the combination of polyorganosiloxane and polyethylene in a softener composition improves the hydrophilicity of fabrics.

The following four softener compositions have been used:

#### *Softener Composition A* (nitrogen-free polyorganosiloxane, Prior Art)

16.7 wt-%	Di(tallowyloxyethyl)(2-hydroxyethyl)methyl ammonium methyl sulfate (90 % solution = 15 wt-% softener)
0.25 wt-%	emulsified <u>nitrogen-free</u> PDMS with 5000 cSt (25% solution = 0.0625 wt-% PDMS)
0.25 wt-%	polyethylene (25 % solution = 0.0625 wt-% polyethylene)
82.8 wt-%	water

#### *Softener Composition B* (nitrogen-containing polyorganosiloxane, Invention)

16.7 wt-%	Di(tallowyloxyethyl)(2-hydroxyethyl)methyl ammonium methyl sulfate (90 % solution = 15 wt-% softener)
0.25 wt-%	emulsified PDMS with 5000 cSt <u>and nitrogen content of 0.0077%</u> (25% solution = 0.0625 wt-% PDMS)
0.25 wt-%	polyethylene (25% solution = 0.0625 wt-% polyethylene)
82.8 wt-%	water

#### *Softener Composition C* (nitrogen-free polyorganosiloxane, Prior Art)

16.7 wt-%	Di(tallowyloxyethyl)(2-hydroxyethyl)methyl ammonium methyl sulfate (90 % solution = 15 wt-% softener)
0.5 wt-%	emulsified <u>nitrogen-free</u> PDMS with 5000 cSt (25% solution = 0.125 wt-% PDMS)
0.5 wt-%	polyethylene (25 % solution = 0.125 wt-% polyethylene)
82.3 wt-%	water

#### *Softener Composition D* (nitrogen-containing polyorganosiloxane, Invention)

16.7 wt-%	Di(tallowyloxyethyl)(2-hydroxyethyl)methyl ammonium methyl sulfate (90 % solution = 15 wt-% softener)
0.5 wt-%	emulsified PDMS with 5000 cSt <u>and nitrogen content of 0.0077%</u> (25% solution = 0.125 wt-% PDMS)
0.5 wt-%	polyethylene (25% solution = 0.125 wt-% polyethylene)
82.3 wt-%	water

ECE 77 standard laundry detergent (ISO 105-C06):

8 % Linear sodium alkyl benzene sulfonate (mean length of alkane chain C11,5)  
2.9 % Ethoxylated tallow alcohol (14 EO)  
3.5 % Sodium soap, chain length (C12-C16: 13 - 26 % C18-C22: 74 - 87 %)  
43.8 % Sodium tripolyphosphate  
7.5 % Sodium silicate ( $\text{SiO}_2/\text{Na}_2\text{O} = 3,3/1$ )  
1.9 % Magnesium silicate  
1.2 % Carboxymethylcellulose (CMC)  
0.2 % EDTA, sodium salt  
21.2 % Sodium sulfate  
9.8 % Water

*Used textile:*

Cotton reinforce (without finishing;  $135 \text{ gm}^{-2}$ ; 5g per sample)

Wash and rinse conditions:

Washing machine: AEG, Ökolavamat 73729

Washing process: short color cycle at  $60^\circ\text{C}$   
20g ECE 77 standard laundry detergent / 1 kg wash load,  
Spin speed 1300 rpm  
Total time: 64 minutes

Wash load: 1 kg fabric

The washing cycle has been repeated three times.

Drying: Tumbler AEG Lavatherm 57719

Afterwards the textile was rewetted with water and 0.059g fabric softener was applied in a LINITEST washing machine (standard laboratory washing machine).

The rinse process:

20°C

5 minutes

liquor ratio 6:1 (total amount of liquid 30g):

Afterwards the fabric is dried at 60°C.

Procedure for measuring the hydrophilicity:

The water absorption of fabrics treated with the softener compositions is measured by the wicking test. The test strips are fixed to a frame and dipped about 1mm deep in a colored aqueous solution. The rise of water in the strips is measured after twenty minutes. The average values of four parallel measurements are given in the following table 1.

Table 1 shows the results of the present comparison test:

	Softener A	<b>Softener B</b>	Softener C	<b>Softener D</b>
Rise of water [mm]	53.5	<b>58.5</b>	53.8	<b>62.3</b>

The fabric treated with the inventive formulation shows an improved hydrophilicity of between 9.3 and 15.8 %.

This behavior could not be expected by a person having ordinary skill in the art.

I, Mario Dubini, finally declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 101 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 6<sup>th</sup>. day of April 2004

A handwritten signature in black ink, appearing to read "Mario Dubini", with a long horizontal flourish extending to the right.

Mario Dubini